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(54) Title: AN AGENT FOR PROTECTION AND LUBRICATION OF PIPE CONNECTIONS, PARTICULARLY PIPE THREAD SECTIONS, AND TUBING ON WHICH SUCH AN AGENT IS APPLIED

(57) Abstract

An agent for protection and lubrication of pipe connections, particularly pipe thread sections on tubing for use in oil and gas wells. This agent comprises a mixture of (a) a grease based component and (b) a solid component free of heavy metal, comprising a material in powder form containing titanium oxide (TiO_2-containing material) having a hardness in the range of from about 6 to about 7 mohs.

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AN AGENT FOR PROTECTION AND LUBRICATION OF PIPE CONNECTIONS, PARTICULARLY PIPE THREAD SECTIONS, AND TUBING ON WHICH SUCH AN AGENT IS APPLIED.

The present application relates to an agent for protection and lubrication in general, but has particular applicability in connection with the storage and use of pipe connections, particularly pipe thread sections, and will in the following be described in terms of such usage. The invention also relates to tubing on which said agent is applied.

The present agent is especially suited for use on tubular materials intended to be used in connection with the drilling and production of oil and gas in both offshore and onshore formations. The agent may be referred to as a universal agent in the sense that it may be used on all types of tubulars irrespective of the quality of steel in the pipe, and both in storage/transport situations and in operative use in oil/gas wells.

Production of oil and gas is a major industry that has been increasing in recent years, both onshore and offshore. Extraction is carried out mainly by large national and international companies operating in accordance with basically the same technological design.

Drilling, extraction and production put very high demands on the methods that are employed, and involve enormous investments with high requirements both for the materials to be used and for protection, preservation and maintenance.

In the drilling and completion of production wells there are used pipes of various types of materials, such as carbon steel, chrome steel, etc., all of which are equipped with

thread systems of various structures and which on installation are joined and screwed together in accordance with more precisely defined technology. The threaded sections are constructed with tolerances that place the strictest demands on protection. The steel mills producing and delivering the drill pipe, casing and tubing provide protection for these products both internally and externally. It is the thread protection, however, which is the most important for all the parts, and which is given by far the greatest attention. Various types of storage grease are used on external and internal ("pin and box end") threads and thread systems, and these are in addition provided with thread protectors, e.g, in the form of screwed-on steel reinforced plastic sleeves or the like, which are not removed before the pipe is to be used in the field unless such removal is necessary for inspection reasons.

The storage agent, which is in the form of a material having the consistency of lubricating grease, should be capable of withstanding atmospheric disturbances, corrosion preventive, water repellent, temperature resistant, easy to apply and remove, and non-toxic to the environment. The usual procedure is to remove the lubricating grease prior to installation and use of the pipes, with new thread grease being applied in order to meet friction and sealing requirements related to the thread geometry and metallurgical demands.

Thus, today there is usually used one agent for application as protection during storage and transport, and one agent intended to replace the first when the pipes are employed in the field where they are screwed together and lowered into the well. As mentioned above, such agent must meet very stringent requirements with regard to performance and temperature resistance. Hence, the API (American Petroleum Institute) has established norms which should be met by said agents, cf. API Bulletin 5A2:

(i) Adequate lubricating properties to prevent tearing (galling) in the thread connections during make-up.

(ii) No tendency to disintegrate or undergo radical change in volume at temperatures up to 150°C,

(iii) No tendency to assume an excessively runny consistency at temperatures as high as 150oC.

(iv) Sealing properties shall be sufficient to prevent leakage at temperatures as high as 150°C.

(v) Absence of any detrimental instability and of any drying or hardening agent which would evaporate or oxidize and thereby alter the properties of the thread paste.

(vi) Resistance to water absorption.

(vii) Sufficient inert filler to prevent leakage in API pipe thread connections at pressures as high as 689.5 MPa.

(viii) Readily applicable with a brush on pipe joints in cold weather.

(ix) Acid resistant in threads and seals.

Previously known lubricating and storage agents for such thread sections comprise a so-called grease base which may also contain silicon compounds, where the grease base consists of a special soap based material together with a process oil. The presence of silicons improves the low temperature properties and can enhance the application on threads that are moistened with water. Silicons do not necessarily improve the sealing or anti-galling properties of a thread compound.

Currently and previously employed agents comprise two component categories, namely (i) a grease based portion and (ii) a solid portion. An example of a known and currently used agent of this type is one consisting of a grease base (in an amount of 36 weight % of the total mixture), graphite in powder form, lead powder, zinc dust and copper flake (in a total amount of 64 weight % of the total mixture). The latter three solid components are thus made up of heavy metals (cf. Bulletin of Thread Compounds for Casing, Tubing and Line Pipe, API Bulletin 5A2 (BUL 5A2), 6th edition, May 31, 1988). With the steadily increasing attention being directed toward the preservation and purification of the environment and the importance of preventing further environmental contamination, it is clear that agents such as those containing heavy metals represent a major disadvantage due to the fact that such heavy metals may have toxic effects on the environment.

It is obvious that the use of lubricating paste containing heavy metals on the threaded sections of drilling/production pipe represents a potential risk of environmental pollution, and may also be injurious for the personnel who are handling such agents.

Also previously known is an agent comprising a grease based component and a solid component of ceramic additives. This agent, however, has been found unsuitable with respect to the requirements established for such agents.

The purpose of the present invention is to provide an agent having storage and lubricating properties for use on pipe thread sections, i.e., an agent having the dual function of serving as a preservative agent during storage and transport and as a lubricating agent when the pipe sections are screwed together. This is in contrast to previously known lubricating agents which do not have properties rendering them suitable for storage purposes, and vice versa.

A second purpose of the invention is to provide an agent having preservative, lubricating and anti-galling properties for use on threaded sections of drilling and production pipes, which does not contain toxic and environmentally harmful heavy metals.

Still another purpose of the invention is to provide an agent of the aforementioned type which, as a minimum, meets the API requirements stated above. It should be added, however, that due to the consideration of attaining optimal, i.e., improved anti-galling effect (as mentioned under (i) above), we have chosen to deviate slightly from the existing API specifications.

Hence, according to the present invention there is provided an agent for protection and lubrication of pipe connections, particularly pipe thread sections on tubing for use in oil and gas wells, and this agent is characterized in that it comprises a mixture of:

(a) a grease based component, and

(b) a solid component free of heavy metal, comprising a material in powder form containing titanium oxide (TiO_2-containing material) having a hardness in the range of from about 6 to about 7 mohs.

It may be mentioned that it is known to use solid filler powders as additives in lubricating grease to minimize wear of bearings under heavy load conditions. Among such additivies are graphite, molybdenum disulphide, asbestos, talc and zinc oxide. Zinc oxide would not be appropriate for use in the present agent because it is undesirable due to contaminating effect on the environment, and because its hardness is unsuitable.

The present agent meets all the above stated requirements (i)-(viii) according to API, although with the exception of the aforementioned deviation in connection with the anti-galling effect. It should be noted, in particular, that the agent is temperature resistant up to above 325°C, which is quite remarkable.

According to the present invention, there is also provided a tubular material that is characterized in that the present agent, as indicated above, is applied to the pipe thread sections and/or connector sections thereof.

A suitable grease based component (a) has the following composition: petroleum sulphonates and calcium sulphonates in an amount of about 80-95 weight % and in particular about 90 weight %, and paraffinic mineral oil in an amount of about 5-20 weight % and in particular 10 weight %.

A special advantage relative to previously known agents that may be mentioned is that the present agent does not dry out (crack), which contributes toward the favorable storage and lubricating properties described above.

Since the grease based portion does not contain any solvents, this emphasizes the environmentally favorable properties of the present product.

The paraffinic mineral oil that is part of component (a) has a function as a softener, is a viscosity regulating agent and provides lubricating properties. Particularly preferred is a grease base sold under the trademark "Mercasol 630" and containing components of the aforementioned type. More specifically, "Mercasol 630" contains calcium petroleum sulphonate (e.g., a commercially available material having the trademark SACI 200A) in an amount of about 90-95 weight % and mineral oil as regulating agent for viscosity in an amount of about 5-10 weight %.

Altogether, the grease base also provides a very good corrosion inhibiting effect, which naturally is of considerable importance in the milieu where the present agent will be used, inter alia in contact with corrosive salts in sea water and subterranean formations.

As the solid component (b) there is used, as mentioned, a material containing titanium dioxide (TiO_2-containing material) having a hardness in the range of from about 6 to about 7 mohs. It should be mentioned that it is also possible to use silicate-containing rock or mineral materials as the solid component (b), such as, for example, biotite ("micromica"), but this would result in the final compound's having properties which represent too great a risk of galling for the metal in the tubular material and is therefore not preferred for all pipe dimensions.

As will be apparent, the solid component (b) contains no heavy metals, and therefore meets today's requirements in the environmental sector. [Paris Commission (PARCOM) Guidelines].

The TiO_2-containing material is preferably made up of pure TiO_2 or the naturally occurring form rutile. Further, it is preferably employed in a particle size in the range of 0-40 micrometers (μm), and most preferably in the range of 0-20 micrometers, and has a hardness in the range of from about 6 to about 7 mohs.

The steel types employed in the actual pipes on whose threads the present agent used have a hardness which lies at about 5.5 mohs. With the use of said TiO_2-containing material having a hardness from about 6 to about 7 mohs, this solid component in the present agent will then give a hardness below the above indicated value for steel, which is of course an absolute requirement.

An embodiment of the present agent comprises about 80-99.9 weight % of grease base and about 0.1-20 weight % of TiO_2-containing material. An especially preferred agent according to the invention comprises about 97 weight % of grease base of the type "Mercasol 630", and about 3 weight % of TiO_2-containing material of the type rutile having a hardness of about 6.5 mohs.

When the present agent is employed on pipe threads, said particulate mineral material will, when the pipes lengths are screwed together ("make-up"), be crushed down into even finer particle sizes which will not disturb the metallic seals achieved between the pipes. In addition, these will give a polishing effect during this screwing-together/make-up oper-

ration, which will also povide a favorable function in connection with unscrewing or disassembly, i.e., when the pipes are for some reason to be loosened/screwed apart again ("break-out"). Thus, the particulate mineral material that is used does not merely contribute filler properties to the present agent.

From this it will be apparent that the mineral, non-toxic additives have a very favorable effect with respect to endowing the present agent with the necessary frictional properties that are so important for the make-up and disassembly operations, i.e., with regard to the torque that must be applied in such operations. The frictional properties must be such that there is no risk of the pipe lengths loosening or becoming unscrewed due to external stresses such as, for example, vibrations in the ground or the formation, mechanical shocks or knocks, etc.

It is common for lubricating grease compositions to undergo an aging process, but said particulate mineral additives do, however, have the favorable characteristic of severely retarding or inhibiting such an aging process and giving the grease base a prolonged and advantageous durability. It has thus been found through experimentation that when pipe threads to which the present agent is applied are screwed together, the torque utilized is the same as the torque required when the pipe threads are screwed apart after an extensive running time. Said experimentation has thus given the following results for the present agent, i.e., a grease base to which a TiO_2-containing material is added:

(a) Torque applied in screwing the sections together (make-up)	4,210 N·m (3,105 ftlbs)
(b) Torque applied in unscrewing the sections (break-out)	4,176 N·m (3,080 ftlbs)
i.e., difference in percent:	0.81 %

Thus, the torque applied in unscrewing/loosening (break-out) is approximately the same as that used during make-up.

In the above experiment as well as in all of those cited below there was used an agent containing about 97 weight % of "Mercasol 630" and about 3 weight % of TiO_2 having a hardness of about 6.5 mohs.

To illustrate how the mineral additives used have a favorable effect on the aging process, i.e., inhibit it, a comparison was made using the grease base alone, i.e., without the mineral additives:

(a) Torque applied in screwing the sections together (make-up)	5,667 N·m (4,180 ftlbs)
(b) Torque applied in unscrewing the sections (break-out)	2,278 N·m (1,680 ftlbs)
i.e., difference in percent:	59.8 %

Thus, the torque related to break-out is considerably less than that for make-up, and this could result in the self-loosening or unscrewing of the pipe lengths, as mentioned above. In practice, the difference between the torque on make-up and on break-out should lie within a tolerance of ± 10 %.

In summary, it can thus be established that the present agent containing said TiO_2-containing material will provide the following advantageous effects:

(i) environmentally favorable properties, i.e., has no contaminating effects;

(ii) polishing effect when pipe sections are screwed together or unscrewed, i.e., favorable frictional characteristics; and

(iii) contributes properties enhancing storage capability;

(iv) meets the requirements that the difference in tolerance on make-up and break-out should be very slight, and

(v) offers savings compared with known and used agents because it may be used both in storage and operational situations, and it requires a lower frequency of inspection due to improved quality.

(vi) friction factor found to be 1,03 according to API RP 7A1 (for rotary shouldered connections).

Since the present agent is, as mentioned above, applicable both for use in storage and transport situations and under operational conditions in oil and gas wells, comparisons have been made between the grease base (A) "Mercasol 630", used as a component in said agent, and which until now has also been used alone (i.e., without the mineral additives) in said storage and transport situations, and the present agent (B).

In the tables given below there is presented data pertaining to use of the present agent compared with other relevant known agents, obtained through laboratory experiments.

Table 1

Corrosion test

Saltspray according to ASTM 117B, ISO 7253, 1984, the present agent applied in a thickness of 15-200 µm on steel plates sand-blasted to minimum SIS (Swedish standard) SA 2.5

Product	0 hours	300 hours	600 hours
A	Ri 0	Ri 0	Ri4 at 20 µm
B	Ri 0	Ri 0	Ri0.5 at 20 µm

A = grease base alone

B = the present agent

Ri = industrial standard for grading of corrosive attack

0 = no rust

Table 2

Heat resistance; consistency after 1 hour of a coating having a thickness of 2 mm (same base surface as in saltspray test)

Product	100°C	200°C	300°C
A	good	good	good
B	good	good	good (also above 325°C)

Table 3

Comparison of products A og B

Product	Saltspray 600 hours	Heat Resistance 1 hour at 250°C	Consi-tency	Application on threads	Appearance after 600 hours salt-spray
A	2	OK	1	easy	2
B	1	OK	1	easy	1

1 = best

2 = poorest

Experimental conditions:

Saltspray:
ASTM 117B, ISO 7253 1982

Heat resistance:
10 mm wide string of the agent in a thickness of 2 mm. Vertically in an oven for 1 hour.

Consistency/application:
Subjective test: Visual impression

Conclusion:
Product B (present agent) shows better overall results than product A (grease base alone). Both products give very good heat resistance and ease of application. No instability in product B, which contains mineral friction components that surprisingly upgrade resistance and stability in relation to corrosion and temperature.

In Table 4 below are given experimental results obtained through laboratory tests for comparison of properties of the present agent against API requirements in accordance with the description under Bulletin 5A2. The testing was carried out in conformity with API specifications pertaining to:

1. Penetration (ASTM D 217) of the agent into metal
   (a) Unused/in use at 25°C (NLGI* Grade No. 1)
   (b) after cooling at -17.8°C

*NLGI = National Lubricating Grease Institute, 4638 J.C. Nichols Parkway, Kansas City 12, Missouri, USA

This test measures the agent's consistency on threads.

2. Mettler Dropping Point,
(ASTM D 566)

This test measures the temperature at which the agent begins to drop.

3. Evaporation, %
24 hours at 100°C

This test measures loss of volatile materials from the agent at 100°C under static conditions.

4. Oil Separation, %
24 hours at 65.6°C

This test measures the tendency of the agent to separate at 65.6°C under static conditions.

5. Water Leaching
2 hours at 65.6°C

This test relates to the determination of the agent's resistance when struck by water.

6. Gas Evolution
65.6°C

This test measures gases that are developed by the agent.

5. Brushing Ability at -17.8°C
(a) Bristle length 42 mm
(b) Bristle length 25 mm
(c) Bristle length 15 mm

This test relates to determination of whether the agent can be applied effectively on threads by use of a brush.

Table 4

Tests	Present agent	API specification
1. Penetration		
(a)	314/319	310/340
(b)	134	200 minimum
2. Dropping Point, °C	> 300	88°C minimum
3. Evaporation, %	0.3	2 maximum
4. Oil Separation, %	1.0	5 maximum
5. Water Leaching	3.1	5 maximum
6. Gas Evolution 65.6°C cm^3	3.4 cm^3	20 maximum
7. Brushing Ability		
(a)	not applicable	applicable
(b)	applicable	applicable
(c)	applicable	applicable

These results illustrate the invention's advantageous properties, and they must thus be viewed in the light of the fact that the present agent does not contain heavy metals.

In the following are reported results obtained through experiments and testing of the present agent and known agents in field tests. The tests have been carried out at Statoil's base in Florø, Norway, using a Weatherford machine. The temperature for the tests was 12-14°C, relative humidity was about 70%, and the weather was good.

The purpose was to test the present agent, which does not contain heavy metals such as lead, copper, zinc, etc., but which still maintains the standards required in API Bulletin 5A2, and to compare this agent with known agents containing

and not containing heavy metals. The present agent, as illustrated in Tables 1-4, has already been tested in comparison with leading types of thread grease, both for storage and operation, where with regard to storage in particular, the emphasis has been placed on:

1) Corrosion resistance
2) Heat resistance
3) Lubricating effect
4) Water displaceability
5) Temperature resistance
6) Application properties
7) Removal properties
8) Non-toxic to the environment

To these are added the most important factors in connection with operation (make-up and break-out situation):

9) Anti-galling effect
10) Non-drying effect without addition of heavy metals
11) Sealing effect

The present agent has shown in all tests that it is very favorable with respect to points 1) - 8). It was therefore of interest to compare the product with the known, commonly used agents with respect to points 9) - 11), with the aid of a field rig test, and to determine the torque relationship between screwing and unscrewing (make-up and break-out).

Basically, two qualities of steel were used, namely

- carbon steel (L-80, N-80 and K-55)
- chrome steel (most commonly 13% chrome).

All pipes having different steel dimensions and steel qualities as well as various thread types were screwed

together according to established torque forces (make-up) measured in ftlbs.

In the test the following types of thread grease were used for testing friction-producing effect and lubricating effect:

Product A Present agent (without addition of heavy metals, but containing mineral material-component (b))

Product B API Mod. Lube Seal, Liquid-O-Ring, with heavy metals (known agent)

Product C Liquid-O-Ring 104, teflon based, without heavy metals (known agent).

The following pipe dimensions were used:

Test 1

5 1/2" - L 80 carbon steel - BDS threads
5 1/2" - 13 CR - 13% chrome steel - BDS threads (thread type from Mannesmann)

Test 2

13 3/8" - N 80 carbon steel - Buttress threads, usual thread type greater dim., 13 3/8" and over)

Test 3

20" - K 55 carbon steel - Big Omega threads, thread type greater dim., 16" and over, from Mannesmann

Test 4

26" - K 55 carbon steel - Big Omega threads, thread type greater dim., 16" and over, from Mannesmann

Test 1

Carried out in a Weatherford machine: Hydraulic Backing Machine - Foster no. 79

	Make-up N·m (ftlbs)	Break-out N·m (ftlbs)	Min. torque, N·m (ftlbs)	Max. torque N·m (ftlbs)	Damage before N·m (ftlbs)	Damage after N·m (ftlbs)
1.1.) 5 1/2" - L 80 - BDS Product A about 20 g	6494 (4790)	none	5396 (3980)	6806 (5020)	none	none
1.2.) 5 1/2" - L 80 - BDS Product B about 25 g	6562 (4840)	none	5396 (3980)	6806 (5020)	none	none
1.3.) 5 1/2" - 13 CR - BDS Product A about 20 g	6847 (5050)	none	6481 (4780)	10874 (8020)	none	none
1.4.) 5 1/2" - 13 CR - BDS Product B about 25 g	8067 (5950)	none	6481 (4780)	8162 (6020)	none	none

The indicated machine of the type Foster no. 79 has:

(i) manually operated release (which may produce variable torque effect)

(ii) no built-in computer or control for break-out reading, and

(iii) diagram reading providing good indications of frictional characteristics in the start-up phase.

The condition of all threads was shown to be very good after testing.

13 CR sleeves, internal thread section (connections) were copper plated. When the present agent was used, there was a visible annular copper encrustation on the sealing surface, external thread section (pin end), which gave a clear indication of metal-to-metal sealing (important for testing).

The following dimensions were used in tests 2, 3 and 4 below:

13 3/8" old pup joint release, about 52,647 N·m (38,830 ftlbs) - cleaned (old)

20" old pup joint release, about 104,195 N·m (76,850 ftlbs) - cleaned (old)

26" old pup joint release, about 99,110 N·m (73,100 ftlbs) - cleaned (new)

A Hydraulic Power Unit Weatherford Machine CAM-F-28 no. 01 was used.

Test 2

	Make-up N·m (ftlbs)	Break-out N·m (ftlbs)	Min. torque, N·m (ftlbs)	Max. torque, N·m (ftlbs)	Damage before, N·m (ftlbs)	Damage after, N·m (ftlbs)
2.1.) 13 3/8" - N-80 - Buttress Product A about 50 g	15497 (11430)	15592 (11500)	11335/ 15117 (8360/ 11150)	18900 (13940)	none	none
2.2.) 13 3/8" N-80 - Buttress Product B about 75 g	14819 (10930)	16161 (11920)	11335/ 15117 (8360/ 11150)	18900 (13940)	none	none

Test 2.1, satisfactory relationship between make-up and break-out; very similar.

Test 2.2, slightly higher torque, no deviation, very similar. The amount of product B increased by about 50 %, from 50 to 75 g. This is a possible reason for the higher torque.

In both tests the sections were screwed to triangle at 9,300 psi/ft, which was continued up to about 14,778-15,456 N·m (10,900-11,400 ftlbs), in order to match make-up and break-out tolerance with similarity from the triangle mark. (Triangle is the symbol for the contact point on the pipe, which is not to be exceeded).

Test 3

	Make-up N·m (ftlbs)	Break-out N·m (ftlbs)	Min. torque, N·m (ftlbs)	Max. torque, N·m (ftlbs)	Damage before, N·m (ftlbs)	Damage after, N·m (ftlbs)
3.1.) 20" - K 55 - Big Omega Product A 100 g	14697 (10840)	14684 (10830)	19659 (14500)	23727 (17500)	none	none
3.2.) 20" - K 55 - Big Omega Product C 125 g	14751 (10880)	12270 (9050)	19659 (14500)	23727 (17500)	none	none

3.1 showed no deviation in the relationship between make-up and break-out.

3.2 Minus tolerance between make-up and break-out of about 17%. Very low break-out, not satisfactory.

Test 4

	Make-up N·m (ftlbs)	Break-out N·m (ftlbs)	Min. torque, N·m (ftlbs)	Max. torque, N·m (ftlbs)	Damage before, N·m (ftlbs)	Damage after, N·m (ftlbs)
4.1.) 26" - K 55 - Big Omega Product A	39834 (29380)	40742 (30050)	25701 (19000)	29828 (22000)	none	none
4.2.) 26" - H 55 Big Omega Product C	40010 (29510)	29354 (21650)	25701 (19000)	29828 (22000)	none	none

The amount of thread grease is not exact; it is in the range of about 175-200 g for test 4.1.), and about 200 g for 4.2.). The reason for the large deviation for make-up/break-out in test 4.2.) is unknown. However, triangle was exceeded by about 3 mm within tolerance in order to achieve equal make-up torque. Otherwise, the difference in percent for test 4.1) is +3 %, and for 4.2.) -27 %.

Conclusion for tests 1-4

The present agent exhibited a more workable consistency than products B and C. It affords simple, easy and even application. Moreover, dismantling of the connectors was very easy, i.e., the lubricating effect was good, particularly for product A. The uniform application resulted in more controlled usage. The color was white and easy to control against the base surface and against impurities. Cleaning--removal of the thread grease -- was slightly easier with product A than with products B and C. Product A, compared

with products B and C, seems to have usage properties that are independent of the particular steel quality, dimension and thread type used, since none of the tests gave a negative result.

The tests show that product A (the present agent), in addition to good storage properties, also has very good and strong qualifications for the operational situation, i.e., it is a good combined agent for storage and running situations. Such a combination effect is not achieved by the known products B and C.

A typical agent according to the present invention has the following data:

(i)	Solvent	none
(ii)	Heavy metal	none
(iii)	Color	white
(iv)	Viscosity/consistency	7200-9000 mPas/paste
(v)	Density	1100 kg/m^3
(vi)	Dry content, weight %	> 99
(vii)	Dry content, volume %	> 99
(viii)	Drying time	non-drying film
(ix)	Cone penetration at 25°	314/319
(x)	Toxicity	non-toxic
(xi)	Temperature resistance (Dropping point)	> 325°C

For the offshore use of the present agent, the agent must meet specific requirements that have been established by the authorities for purposes relating to environmental safety. The agent has therefore undergone the following ecotoxicological tests at SINTEF (Norway) (according to PARCOM guidelines):

(a) Phytoplancton test (Skeletonema coctstum)

(b) Blue mussel test (Mytilus edulis)

(c) Barnacle test (Balanus improvisus).

The agent has also been subjected to a

(d) Bioaccumulation test

at Aquateam A/S, Norway, according to OECD guidelines no. 117 for testing of chemicals.

These tests (a)-(d) have shown that the agent satisfies the requirements for usage that have been established in accordance with the PARCOM guidelines.

Patent Claims

1.
Agent for protection and lubrication of pipe connections, particularly pipe thread sections on tubing for use in oil and gas wells, characterized in that it comprises a mixture of:

(a) a grease based component, and

(b) a solid component free of heavy metal, comprising a material in powder form containing titanium oxide (TiO_2-containing material) having a hardness in the range of from about 6 to about 7 mohs.

2.
An agent according to claim 1, characterized in that component (a) constitutes a mixture of petroleum sulphonates, calcium sulphonates and paraffinic mineral oil; about 80-95 weight % and in particular about 90 weight % of petroleum and calcium sulphonates, and about 5-20 weight % and in particular about 10 weight % of paraffinic mineral oil.

3.
An agent according to claims 1 and 2,
characterized in that component (a) constitutes a "Mercasol 630", i.e., about 90-95 weight % of calcium petroleum sulphonate and about 5-10 weight % of mineral oil.

4.
An agent according to claim 1, characterized in that the TiO_2-containing material consists of rutile.

5.
An agent according to claims 1-4, characterized

i n that the TiO_2-containing material has a particle size in the range of 0-40, preferably 0-20 µm.

6.
An agent according to claims 1-5, c h a r a c t e r i z e d i n that it comprises:

(i) about 80-99.9 weight % of a grease base
(ii) about 0.1-20 weight % of TiO_2-containing material.

7.
An agent according to claims 1-6, c h a r a c t e r i z e d i n that it comprises:

(i) about 97 weight % of a grease base of the type "Mercasol 630"
(ii) about 3 weight % TiO_2-containing material of the type rutile.

8.
A tubular material, c h a r a c t e r i z e d i n that an agent in accordance with the claims 1-7 is applied to the pipe thread sections and/or connector sections thereof.

AMENDED CLAIMS

[received by the International Bureau on 16 February 1993 (16.02.93); original claims 1-8 replaced by amended claims 1-7 (2 pages)]

1.
The use of a mixture of (a) a grease based component comprising a mixture of petroleum sulphonates, calcium sulphonates and paraffinic mineral oil; about 80-95 weight % and in particular about 90 weight % of petroleum and calcium sulphonates, and about 5-20 weight % and in particular about 10 weight % of paraffinic mineral oil, and (b) a solid component free of heavy metal, comprising a material in powder form containing titanium oxide (TiO_2-containing material) having a hardness in the range of from about 6 to about 7 mohs, as an agent for protection and lubrication of pipe connections, particulary pipe thread sections on tubing for use in oil and gas wells.

2.
The use according to claim 1 wherein component (a) constitutes a "Mercasol 630", i.e., about 90-95 weight % of calcium petroleum sulphonate and about 5-10 weight % of mineral oil.

3.
The use according to claim 1 wherein the TiO_2-containing material consists of rutile.

4.
The use according to claim 1 wherein the TiO_2-containing material has a particle size in the range of 0-40, preferably 0-20 μm.

5.
The use according to claims 1-4, wherein said mixture comprises about 80-99,9 weight % of the grease base, and about 0,1-20 weight % of the TiO_2-containing material.

6.
The use according to claim 5 wherein said mixture comprises about 97 weight % of a grease base of the type "Mercasol 630", and
about 3 weight % TiO_2-containing material of the type rutile.

7.
A tubular material , c h a r a c t e r i z e d in that an agent us used in claim 1 - 6 is applied to the pipe thread sections and/or connector sections thereof.

INTERNATIONAL SEARCH REPORT

International Application No PCT/NO 92/00146

I. CLASSIFICATION OF SUBJECT MATTER (if several classification symbols apply, indicate all)[6]

According to International Patent Classification (IPC) or to both National Classification and IPC

IPC5: C 10 M 125/10

II. FIELDS SEARCHED

Minimum Documentation Searched[7]

Classification System	Classification Symbols
IPC5	C 10 M

Documentation Searched other than Minimum Documentation to the Extent that such Documents are Included in Fields Searched[8]

SE,DK,FI,NO classes as above

III. DOCUMENTS CONSIDERED TO BE RELEVANT[9]

Category *	Citation of Document,[11] with indication, where appropriate, of the relevant passages[12]	Relevant to Claim No.[13]
X	Dialog Informaton Services, file 350, (World Patent Index) Dialog accession no. 001160030, (HITACHI LTD), "Conductive lubricating compsn - contg mineral oil, wax, GPV or VI metal cpds, graphite and metal oxides", JP 74014842, B, 740410, 7418	1,4
Y	--	2-3,5-8
Y	EP, A1, 0182441 (STAUFFER CHEMICAL COMPANY) 28 May 1986, see the abstract	2-3,5-8
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* Special categories of cited documents:[10]

"A" document defining the general state of the art which is not considered to be of particular relevance

"E" earlier document but published on or after the international filing date

"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)

"O" document referring to an oral disclosure, use, exhibition or other means

"P" document published prior to the international filing date but later than the priority date claimed

"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

"X" document of particular relevance, the claimed invention cannot be considered novel or cannot be considered to involve an inventive step

"Y" document of particular relevance, the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.

"&" document member of the same patent family

IV. CERTIFICATION

Date of the Actual Completion of the International Search	Date of Mailing of this International Search Report
14th December 1992	22-12-1992
International Searching Authority	**Signature of Authorized Officer**
SWEDISH PATENT OFFICE	Anita Skeppstedt

ANNEX TO THE INTERNATIONAL SEARCH REPORT ON INTERNATIONAL PATENT APPLICATION NO.PCT/NO 92/00146

This annex lists the patent family members relating to the patent documents cited in the above-mentioned international search report. The members are as contained in the Swedish Patent Office EDP file on 02/12/92
The Swedish Patent Office is in no way liable for these particulars which are merely given for the purpose of information.

Patent document cited in search report	Publication date	Patent family member(s)		Publication date
EP-A1- 0182441	86-05-28	JP-A-	61124537	86-06-12
		US-A-	4604227	86-08-05